United States Department of Agriculture
Agricultural Research Administration
Bureau of Entomology and Plant Quarantine

NICOTINE INSECTICIDES. PART III--DUST CARRIERS FOR NICOTINE

By E. L. Mayer, Division of Control Investigations, Bureau of Entomology and Plant Quarantine, and Florence B. Talley and C. F. Woodward, Eastern Regional Research Laboratory, Bureau of Agricultural and Industrial Chemistry 1/

This is the third of a series of investigations on nicotine insecticides conducted by this Bureau in cooperation with the Bureau of Agricultural and Industrial Chemistry. Part I of this series (E-646 issued in 1945) reported a study of complex salts containing nicotine, usually combined with a metal, and Part II (E-709 issued in 1946) described tests with a large number of materials to find activators for nicotine. The purpose of the present study was to determine the effect of various carriers on the toxicity of dusts or sprays containing nicotine sulfate.

As before, the materials tested were prepared at the Eastern Regional Research Laboratory of the Bureau of Agricultural and Industrial Chemistry, for testing against plant-feeding insects by the Bureau of Entomology and Plant Quarantine at its Sanford, Fla., laboratory.

Previous investigations by Headlee and Rudolfs (3, 4) and by Thatcher and Streeter (9) have shown that there are three types of dust carriers for nicotine or nicotine sulfate: (1) "Adsorbent" carriers, which tend to prevent the volatilization of nicotine: (2) "inert" carriers, which merely expose large surface areas; and (3) "active" carriers, which liberate nicotine from its salts by chemical action. Examples of these types are bentonite, pyrophyllite, and dolomite, respectively. These early studies were concerned largely with the control of aphids, and the active carriers were the most effective of the three types. It was further shown by De Ong (1) and Headlee and Rudolfs (3) that the toxicity of nicotine-containing dusts and sprays was directly proportional to the rate of nicotine evolution.

1/The authors are indebted to E. R. McGovran (transferred to Office of Experiment Stations, November 3, 1946) and G. T. Bottger, Division of Control Investigations, for supervision of the entomological tests; and to J. J. Willaman, Eastern Regional Research Laboratory, for many valuable suggestions in this investigation.

The volatilization of nicotine from dusts was affected by factors other than the type of carrier. Nicotine was more readily lost from a coarse dust than from a fine one (2, 4). In aphid control a temperature of at least 70° F. was required for satisfactory evolution of the alkaloid. Small amounts of water hastened nicotine evolution from the active carriers (4, 7, 9).

In an attempt to increase the efficiency of nicotine dusts against several lepidopterous larvae, it was deemed advisable to investigate in a preliminary manner the effect of various carriers in the dust mixtures. Accordingly 39 materials, selected to represent a wide range in particle size, were obtained from commercial companies in March 1944. Particular attention was directed to carriers that did not release nicotine from the sulfate, inasmuch as a lasting deposit of the toxicant appeared desirable. However, several active carriers were included for comparison. A further objective of these comparisons was to select a carrier which would be used as a standard in a large number of dust mixtures employed in screening tests on nicotine derivatives and in the search for nicotine synergists. Characteristics which must be considered to appraise fully the economic usefulness of carriers such as dustability, drifting, packaging requirements, availability, and standardization were not studied in the laboratory tests. Furthermore, no attempt was made to evaluate any possible synergistic action of the carriers which gave the best results.

This investigation was confined to preliminary observations of the toxicity of the various nicotine dust mixtures to the melonworm (_Diaphania_ _hyalinata_ (L.)) and the southern armyworm (_Prodenia_ _eridania_ (Cram.)).

Materials and Experimental Procedure

All mixtures contained 5 percent of nicotine on the alkaloid basis. In the bentonite this was supplied by free alkaloid; in the others by nicotine sulfate.

The 37 carriers studied, plus the 2 in the standard dusts, Pyrax ABB (pyrophyllite) (No. 38) and Volclay bentonite (No. 39), are listed in table 1. With the exception of Carolina pyrophyllite (No. 19), which was approximately 200 mesh, all dusts were 300-325 mesh or finer.

The pH of the carriers was determined by suspending 5 grams in 20 ml. of water and allowing the suspension to stand until a constant pH value was reached. The pH of the carriers plus 5 percent of nicotine as the sulfate was determined in a similar manner. All readings were made with a glass-electrode pH meter.

The loss of nicotine was determined by chemical analysis of the mixtures before and after 16 hours' exposure at 70° F. In these tests 2 grams of dust mixture was uniformly distributed on a square foot of glass area. Nicotine determinations were made by the silicotungstic acid procedure. The percentages of nicotine lost from the dusts containing 5 percent of nicotine as the sulfate are reported in table 1.

Most of the dusts were tested against the first-instar southern armyworm and the fourth-instar melonworm. The pulverized limestone mixture, which was not of a dustable consistency, was restricted to spray tests.

The testing procedure used was similar to that described by Swingle (8) and in Part I of this series. About 30 larvae were used in each test. The large larvae were placed on previously dusted pumpkin-leaf sections in 9-cm. petri dishes and the small larvae in cloth-covered vials on dusted collard-leaf sections. Counts were taken at the end of the first, second, and third days. The test was terminated on the third day.

A nicotine sulfate dust (nicotine 5 percent) in pyrophyllite (Pyrax ABB) was used as the standard of comparison. Some of the mixtures that gave a higher mortality than the standard were tested in sprays and subsequently compared with nicotine bentonite. This work was not completed, however, because the entomological laboratory was transferred from Sanford, Fla., to Anaheim, Calif., where the melonworm and the southern armyworm were not available.

Discussion of Results

Of the 37 materials tested as carriers for nicotine, 9 gave an increase in toxicity of more than 15 percent over the standard in at least one test (table 2).

When compared with the Pyrax ABB (pyrophyllite) standard, the nicotine dusts that showed the highest mortalities of the southern armyworm were those containing magnesia talc, fuller's earth E 90-44B, talc No. 21, talc No. 23, fuller's earth E 90-44A, and fire clay. Against the melonworm the most effective carriers were Grundite Bond, magnesia talc, and Cherokee clay. Fuller's earths E 90-44A and E 90-44B were slightly above the standard in tests on melonworm. When used in sprays, pulverized limestone, fuller's earth E 90-44A, magnesia talc, Grundite Bond, and fuller's earth E 90-44B gave better results than the standard against the southern armyworm, and magnesia talc gave the best results against the melonworm.

Of the carriers compared with bentonite, fire clay was the only material that showed up as possibly better, both in a dust and in a spray, against the melonworm. Grundite Bond was somewhat better in a dust against the melonworm, while fuller's earth E 90-44A was better in a spray against the southern armyworm. All other materials were inferior to bentonite in most tests. It should be noted that in the bentonite series nicotine sulfate is compared with what is usually considered to be a reaction product, namely, nicotine bentonite (5, 6).

Both acid and alkaline materials are represented in the list of 9 best dust carriers. The clays are essentially acid; the pH of fire clay is 4, of Cherokee clay 6.1, and of Grundite Bond 5.97. The talcs and limestone are alkaline; talc No. 21 has a pH of 8.5, talc No. 23 of 8.55, magnesia talc of 8.95, and pulverized limestone of 8.89. The fuller's earths, E 90-44A and E 90-44B, are somewhat less alkaline than the talcs, having pH values of 7.8 and 8.02, respectively. It is believed that the increased kills of the southern armyworm with some of these alkaline materials, especially limestone, may have been due to nicotine fumigation, as the cloth-covered vials in which the tests were carried out do not afford much ventilation.

Of the 9 best dusts only 2 showed any great loss of nicotine during the 16-hour period of exposure. Pulverized limestone lost 98 percent of its nicotine, which was expected on the basis of previous findings. Talc No. 23 lost 27 percent and talc No. 21 lost 17 percent. The other 6 dusts lost only 1 to 9 percent of nicotine.

It is recognized that the physical characteristics of the dusts may have influenced their effectiveness. The addition of enough nicotine sulfate to give a dust containing 5 percent of nicotine in many cases resulted in a poorly dustable mixture with impaired efficiency.

In this regard special mention should be made of the unique physical properties of fuller's earth E 90-44B, mineralogically known as attapulgite. It is a very light, fluffy material with a remarkable power to absorb liquids. As much as 33 percent of mineral oil has been incorporated in this material without impairing its excellent dusting qualities. On the basis of these preliminary data we suggested that attapulgite be considered as a carrier for insecticides. 2/

Summary

Thirty-seven materials were tested as carriers for nicotine sulfate against the melonworm (*Diaphania hyalinata* (L.)) and the southern armyworm (*Prodenia eridania* (Cram.)). Nine materials appeared to

---

2/Since this manuscript was submitted, attapulgite has found extensive commercial use as an insecticide carrier.

produce higher kills than the pyrophyllite standard in at least one test. Three of them, magnesia talc and fuller's earths E 90-44A and E 90-44B, gave considerably higher kills against both insects in both dusts and sprays.

Of the limited group compared with the bentonite standard, fire clay was as good or better, both in dusts and sprays, against the melonworm. Fuller's earth E 90-44A was better in dusts against the melonworm, and fuller's earth E 90-44B was better in sprays against the southern armyworm.

The two standards were compared with each other in only one spray test, and in this case bentonite was better than the pyrophyllite (Pyrax ABB) against the armyworm.

Insect mortality appeared to have no well-defined relation to pH or to nicotine volatility, since acid and alkaline materials were represented among the 9 best materials, while only 2 of them showed appreciable nicotine loss.

Fuller's earth E 90-44B, or attapulgite, is considered the best of the new materials tested because of its unique ability to absorb large quantities of liquid, either oil or water, and because of the high mortalities resulting from its use.

Table 1.--pH values and nicotine retentiveness of dusts used as carriers.

	Carrier	Supplier 1/	pH found	pH of carrier + 5 percent of nicotine	Nicotine lost after 16 hours' exposure
					Percent
1	Colloidal kaolin	United Clay Mines, Trenton, N. J.	4.4	5	14
2	Harmon clay	do.	4.3	5.6	0
3	Peerless clay	R. T. Vanderbilt Co., 230 Park Ave., New York, N. Y.	5.5	5.4	8.6
4	Fillmore clay	United Clay Mines	4.5	5.9	11.4
5	Fire clay	Illinois Clay Products Co., Joliet, Ill.	4.0	4.7	9.4
6	Perry clay	United Clay Mines	4.1	5.1	3.2
7	Cherokee clay	R. T. Vanderbilt Co.	6.1	6.4	3.0
8	Bancroft clay	United Clay Mines	4.1	5.2	11.5
9	Topton clay	do.	4.6	5.1	16.3
10	Chicora clay	J. J. Huber, Inc., 460 W. 34th St., New York, N. Y.	4.6	4.9	0
11	Gypsum	National Gypsum Co., York, Pa.	7.3	6.6	20.3
12	Walnut shell flour	Agicide Laboratories, 4668 N. Teutonia Ave., Milwaukee, Wis.	4.8	5.0	12.6
13	Talc No. 21	W. H. Richards Co., 1203 E. State St., Trenton, N. J.	8.5	6.9	16.6
14	Talc No. 23	Eastern Magnesia Talc Co., Burlington, Vt.	8.6	7.6	27.1

Table 1.--Continued

	Carrier	Supplier 1/	pH found	pH of carrier ≠ 5 percent of nicotine	Nicotine lost after 16 hours' exposure
					Percent
15	Fibrous talc (Loomkill)	Loomis Talc Co., Gouverneur, N. Y.	9.1	8.4	63.2
16	Magnesia talc	Eastern Magnesia Talc Co.	9.0	7.1	1.8
17	Velvet filler R	Carbola Chemical Co., Natural Bridge, N. Y.	9.1	8.7	62.1
18	Micro Velva A	do.	9.0	8.8	45.5
19	Carolina pyrophyllite	Carolina Pyrophyllite Co., 10 E. 40th St., New York, N. Y.	6.9	5.1	3.1
20	Grundite Bond	Illinois Clay Products Co.	6.0	6.5	1.7
21	Fuller's earth E 90-44A	Attapulgus Clay Co., Philadelphia, Pa.	7.8	7.2	3.7
22	Fuller's earth E 90-44B	do.	8.0	7.6	1.2
23	Bauxite E 90-44C	do.	6.5	6.3	18.4
24	Frianite M3X	Butcher and Co., Los Angeles, Calif.	6.3	6.0	24.4
25	Frianite DS	do.	6.5	6.0	35.7
26	Wyobond bentonite	Wyodak Chemical Co., 4600 E. 71st St., Cleveland, Ohio	8.8	8.6	0
27	Volclay bentonite (Hivo)	American Colloid Co., 362 W. Superior St., Chicago, Ill.	8.8	8.4	0
28	Volclay bentonite (Visclo)	do.	8.6	8.1	0

Table 1.--Continued

	Carrier	Supplier 1/	pH found	pH of carrier + 5 percent of nicotine	Nicotine lost after 16 hours' exposure
					Percent
29	Silene EF Batch 3-7-1	Pittsburgh Plate Glass Co., Columbia Chem. Div., 30 Rockfeller Plaza, New York, N.Y.	8.8	8.8	0
30	Micronized Pyrax ABB	do.	5.9	4.3	2.0
31	Ferasil (Fly Ash)	Corson Lime Co., Plymouth Meeting, Pa.	8.2	7.3	0
32	No. 2261 Florida land-pebble super-phosphate	Baugh Co., Philadelphia, Pa.	2.9	3.1	11.0
33	Diluex	Floridin Co., Warren, Pa.	7.9	7.0	9.5
34	Florigel	do.	7.5	6.9	0
35	China clay	Wagner Co., Philadelphia, Pa.	7.8	7.5	30.5
36	Pulverized limestone	Corson Lime Co., Plymouth Meeting, Pa.	8.9	8.3	98.3
37	Magnesite	do.	9.2	8.2	74.1
38	Pyrax ABB	R. T. Vanderbilt Co.	7.6	6.6	6.0
39	Volclay Wyoming bentonite	Wagner and Co.	9.1	---	0

1/The address of each supplier is given only once in this table.

Table 2.--Percentage increase or decrease in toxicity of nicotine dusts or sprays containing carriers that in some tests were at least 15 percent more toxic than nicotine insecticides containing a standard carrier

	Carrier	Comparison with pyrophyllite standard				Comparison with bentonite standard		
		Dust		Spray		Dust	Spray	
		Southern armyworm	Melon-worm	Southern armyworm	Melon-worm	Melon-worm	Southern armyworm	Melon-worm
5	Fire clay	+ 17	+ 8	- 3	+ 5	+ 6	--	+ 16
7	Cherokee clay	- 13	+ 16	--	--	--	--	--
13	Talc No. 21	+ 19	- 13	--	--	--	--	--
14	Talc No. 23	+ 18	- 6	--	--	--	--	--
16	Magnesia talc	+ 34	+ 19	+ 26	+ 21	- 27	--	- 52
20	Grundite Bond	- 20	+ 22	+ 24	--	+ 10	- 10	- 22
21	Fuller's earth E 90-44A	+ 17.5	+ 10.5	+ 52	--	- 5	+ 18	- 13
22	Fuller's earth E 90-44B	+ 29.5	+ 14	+ 16	--	- 6	- 18	--
36	Pulverized limestone	--	--	+ 56	- 22	--	--	--

Table 3.--Materials comparable to or poorer than the standard in most tests

	Carrier	Southern armyworm	Melon-worm
1	Colloidal kaolin	+ 1/	-
2	Harmon clay	+	-
3	Peerless clay	-	+
4	Fillmore clay	+	-
6	Perry clay	+	-
8	Bancroft clay	-	-
9	Topton clay	-	-
10	Chicora clay	-	-
11	Gypsum	+	-
12	Walnut shell flour	-	-
15	Fibrous talc (Loomkill)	-	-
17	Velvet filler R	-	-
18	Micro Velva A	-	+
19	Carolina pyrophyllite	+	+
23	Bauxite E 90-44C	-	-
24	Frianite M3X	-	-
25	Frianite DS	-	-
26	Wyobond bentonite	-	+
27	Volclay bentonite (Hivo)	-	-
28	Volclay bentonite (Visclo)	-	-
29	Silene EF	+	-
30	Micronized Pyrax ABB	-	-
31	Ferasil (Fly Ash)	-	-
32	No. 2261 Florida land pebble superphosphate	-	-
33	Diluex	-	-
34	Florigel	-	+
35	China clay	-	-
37	Magnesite	-	-

1/ + Slightly better than standard; - poorer than standard.

Literature Cited

(1) De Ong, E. R.
1923. The relation between the volatility and toxicity of nicotine in sprays and dusts. Jour. Econ. Ent. 16: 486-493.

(2) ____________
1943. Frianite, an insecticide diluent. Jour. Econ. Ent. 36: 799-800.

(3) Headlee, T. J., and Rudolfs, W.
1923. Some principles which underlie the making and use of nicotine dust. N. J. Agr. Expt. Sta. Bul. 381, 47 pp.

(4) ____________ and Rudolfs, W.
1924. Some further facts relative to the principles underlying the making and use of nicotine dust. N. J. Agr. Expt. Sta. Bul. 400, 44 pp.

(5) Smith, C. R.
1934. Base exchange reactions of bentonite and salts of organic bases. Amer. Chem. Soc. Jour. 56: 1561-1563.

(6) ____________
1937. Compound and process for making nicotine-bentonite compounds. (U. S. Patent No. 2,096,566) U. S. Patent Office, Off. Gaz. 483: 649.

(7) Streeter, L. R.
1925. Influence of temperature and humidity upon the volatilization of nicotine from tobacco dust-lime hydrate mixtures. Jour. Econ. Ent. 18: 590-593.

(8) Swingle, M. C.
1943. Exploring the insecticidal possibilities of new materials. In Laboratory procedures in studies of the chemical control of insects, edited by F. L. Campbell and F. R. Moulton, Amer. Assoc. Adv. Sci., Pub. 20, pp. 82-84. Washington, D. C.

(9) Thatcher, R. W., and Streeter, L. R.
1923. Factors which affect the volatility of nicotine from insecticide dusts. N. Y. Agr. Expt. Sta. Bul. 501, 34 pp.